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ESTIMATION OF SHELL LIFE
OF
UNSTABILIZED POLYOLEFINS
FROM
THERMAL OXIDATION DATA

LEO REICH

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PICATINNY ARSENAL
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ESTIMATION OF SHELF LIFE OF
UNSTABILIZED POLYOLEFINS FROM
THERMAL OXIDATION DATA

by
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OBJECT

To estimate the shelf life of unstabilized polyolefins from the extrapolation of carbonyl formation data obtained during the thermal oxidative degradation of such polyolefins.

SUMMARY

Utilizing carbonyl formation data previously obtained during the thermal oxidation of unstabilized atactic polypropylene (APP), un-stabilized isotactic polypropylene (IPP) and unstabilized atactic poly-butene-1 (APB) along with a semi-empirical expression, it was possible to extrapolate and obtain arbitrary induction times (θ) at relatively low temperatures. Values of θ thus obtained were correlated with changes in physical properties, i.e., intrinsic viscosity and the product of ultimate elongation (UE) and ultimate tensile strength (UTS) (a product which is often referred to as the impact index).

INTRODUCTION

Numerous attempts have been made to correlate outdoor weathering of plastics with accelerated aging (e.g., Ref 1). Generally, such correlations have been reported to be poor due to the many factors involved in the natural and artificial photochemical aging processes. Besides photochemical oxidation, various workers have employed thermal oxidation.

Thus, Grieveson and coworkers (Ref 2) investigated the effect of air oxidation of unstabilized high density polyethylene at 120° C on physical properties. They indicated that, although very little oxygen had been absorbed at the end of the induction period, considerable changes in intrinsic viscosity and flex value had occurred. For all practical purposes, therefore, it seemed that at the end of the induction period the polyethylene sample had reached also the end of its useful life. Oswald and Turi (Ref 3) studied the effect of oxidative degradation on the deterioration of physical properties of unstabilized isotactic polypropylene (in 100% oxygen at 75° C). They found that the absorption of only 1.1 mg oxygen per gram of IPP resulted in a 20% loss in properties, e.g., in the impact index. Further, these workers indicated that extrapolation of elevated temperature data to ambient temperatures was unsafe (as judged by changes in physical properties). Thus, utilizing an apparent activation energy of 30-32 kcal-mole^{-1}, a life expectancy of 6 years for IPP in air at 25° C was calculated as compared with the experimental value of approximately 16 months (based on viscosity changes).

The purpose of this report is to present a semi-empirical expression which, in conjunction with carbonyl formation data at elevated temperatures, may be used for extrapolation to obtain useful lifetime values of unstabilized polyolefins (APP, IPP, and APB) at ambient temperatures.

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RESULTS AND DISCUSSION

Based on theoretical considerations previously presented (e.g., Ref 4), the following expression may be written for rate of carbonyl formation, ρ_{co}, during polyolefin oxidation:

$$\rho_{co} \approx C\ R_i t \qquad\qquad (1)$$

where $C = k_3 k_6 / (k_3 + k_4)$; k's denote rate constants; t = times; and R_i denotes initiation rate and is equal to k_i [RH] [O_2] where, in turn, k_i = initiation rate constant, [RH] = concentration of reactive hydrogen on polymer main chain, and [O_2] = oxygen concentration in percent. [In the derivation of Equation 1, it was assumed that the term At << 1 (see Ref 5)] . Upon integrating Equation 1, there is obtained

$$[CO] = C\ k_i\ [RH]\ [O_2]\ \frac{t^2}{2} \qquad\qquad (2)$$

Now, if we assume that at $t = \Theta$ (an induction time) Equation 2 is still valid, this equation becomes,

$$[CO]_{ind} = C\ k_i\ [RH]\ [O_2]\ \frac{\theta^2}{2} \qquad\qquad (2a)$$

where $[CO]_{ind}$ = carbonyl concentration during induction time θ. When $[CO]_{ind}$ is maintained constant, the Arrhenius relation is employed, and it is assumed that $(k_3 + k_4)/k_3$ is approximately constant at various temperatures (see Ref 6) then,

$$\ln\left\{ \theta^2\ [O_2] \right\} = \ln K + (E_i + E_6)/RT \qquad\qquad (3)$$

where $K = 2\ [CO]_{ind}\ (k_3 + k_4)/k_3\ [RH]\ Z_i\ Z_6$; Z's are frequency factors; E's denote activation energies; R = gas constant; and T = temperature.

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In order that $[CO]_{ind}$ be approximately constant in Equation 3, we arbitrarily let $\theta = 0.4\,t_m$ where t_m = time at which the maximum rate of carbonyl formation is reached (see Fig 1). At this value of θ, $[CO]_{ind}$ should be very low and approximately equal at various values of $[O_2]$ and temperature. Further, at this value of θ, the deterioration of physical properties of the polymers may not be too severe as yet.

In Figures 1 through 7 are depicted plots of carbonyl absorbance area (from infrared measurements and in arbitrary units (see Ref 7 through 9) vs time for the oxidation of APP and IPP at various temperatures and oxygen concentrations. From such typical plots were obtained values as presented in Tables 1 through 3 for APP, IPP, and APB. In Figures 8A and 8B are shown plots of average values of $\log(\theta_2\,[O_2])$ versus $\dfrac{1}{T}$ (reciprocal temperature) for APP, IPP, and APB. In the plots for APP and IPP (Fig 8A), the derived data virtually coincide. (This may be due to the equal reactivity of amorphous regions in APP and IPP, which results in a similar time interval for obtaining maximum carbonyl formation.) Using the mean deviations for values of $\log(\theta^2\,[O_2])$, upper and lower limits were drawn (Fig 8A) for the data, and the intermediate linear relation was obtained from a least squares analysis (the linear correlation coefficient possessed a value between 0.98 - 0.99). The values of the apparent activation energies (see Eq 3) (going from the lower limit to the upper limit) were: 32, 37, and 42 kcal-mole^{-1}.

From the lower limit plot a value of $\theta = 6.3$ hours can be calculated for $[O_2] = 100\%$ and 75°C. Data of Oswald and Turi (Fig 8, 11, 12, and 13 of Ref 3) indicated that at 75°C and $[O_2] = 100\%$ and $\theta = 6.3$ hours, the change in intrinsic viscosity was relatively small, and there was 85-100% retention in the impact index and in the ultimate tensile strength and ultimate elongation of the unstabilized polypropylene film used (which was of 25 mil thickness). Further, from the lower limit and the linear regression line (Fig 8A), the values of θ were, respectively, 50 and 100 days at 22°C, and $[O_2] = 20\%$. Oswald and Turi observed that marked

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changes in intrinsic viscosity commenced when unstabilized polypropylene powder was shelf-stored at ambient temperature in air for approximately 450 days. In this respect, it may be remarked that relatively large changes in mechanical properties undoubtedly occurred long before the marked change in intrinsic viscosity took place (see Fig 8 and 12 of Ref 3). Thus, the lower limit line may provide a fairly safe prediction of the maximum time that unstabilized polypropylene should be shelf-stored (or maintained at higher temperatures) before its mechanical properties would degrade considerably. However, it should be noted here that much more data on shelf-life stability of unstabilized polypropylene is needed before any definite conclusions can be drawn.

In Figure 8B is depicted a plot of log ($\theta_2 \left[0_2 \right]$) vs $\frac{1}{T}$ for APB.

The lower limit and least square lines afforded values of activation energies of, respectively, 46 and 55 kcal-mole^{-1} (a value of $E_i + E_6 = 43$ kcal-mole^{-1} has been reported (Ref 10). However, due to lack of appropriate data on unstabilized APB shelf-life, no extrapolations to ambient temperature were made. Nevertheless, it would appear from a comparison of Figures 8A and 8B that un-stabilized APB would provide a longer shelf life than APP or IPP.

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TABLE 1
Data for autoxidation of APP

Temperature °C	$\frac{1}{T} \times 10^3$ °K^{-1}	t_m hr	θ^2 hr^2	$[O_2]$ %	$\log(\theta^2 [O_2])$ hr^2
110	2.611	5.0	4.00	10	1.60
		5.0	4.00	20	1.90
		4.5	3.24	50	2.21
		4.6	3.38	100	2.53
					Avg 2.06 ± 0.31
120	2.545	3.5	1.96	5	0.99
		3.5	1.96	10	1.29
		3.3	1.74	20	1.54
		2.5	1.00	75	1.88
		2.2	0.78	100	1.89
					Avg 1.52 ± 0.30
130	2.481	3.0	1.44	5	0.86
		2.8	1.25	10	1.10
		1.8	0.52	20	1.02
		1.6_6	0.44	50	1.34
		1.2	0.23	75	1.24
		1.2	0.23	100	1.36
					Avg 1.15 ± 0.16

TABLE 2
Data for autoxidation of IPP

Temperature °C	$\frac{1}{T} \times 10^3$ °K^{-1}	t_m hr	θ^2 hr^2	$[O_2]$ %	$\log(\theta^2 [O_2])$ hr^2
120	2.545	3.5	1.96	11	1.35
		3.0	1.44	20	1.46
		2.0	0.64	50	1.51
		1.7	0.46	75	1.54
		1.5	0.36	100	1.56
					Avg 1.48 ± 0.064
130	2.481	2.5	1.00	7	0.85
		2.3	0.85	11	0.97
		1.7	0.49	50	1.39
		1.5	0.36	75	1.43
					Avg 1.16 ± 0.25
140	2.421	1.5	0.36	7	0.40
		1.2	0.23	20	0.66
		0.80	0.10	50	0.70
		0.66	0.070	75	0.72
		0.50	0.040	100	0.60
					Avg 0.62 ± 0.090
150	2.364	1.0	0.16	7	0.049
		0.75	0.090	11	0.00
		0.63	0.064	20	0.11
		0.25	0.010	100	0.00
					Avg 0.040 ± 0.04

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TABLE 3
Data for autoxidation of APB

Temperature $^{\circ}C$	$\frac{1}{T} \times 10^3$ $^{\circ}K^{-1}$	t_m hr	θ^2 hr^2	$[O_2]$ %	$\log(\theta^2[O_2])$ hr^2
110	2.611	10.0	16.0	50	2.90
		7.7	9.49	75	2.85
		4.5	3.24	100	2.51
					Avg 2.75 ± 0.16
115	2.578	7.8	9.73	25	2.38
		5.5	4.84	50	2.38
		5.0	4.00	75	2.48
		2.7	1.17	100	2.07
					Avg 2.33 ± 0.13
120	2.545	5.6	5.02	12.5	1.79
		3.6	2.10	25	1.72
		2.4	0.92	75	1.84
		1.4	0.31	100	1.49
					Avg 1.71 ± 0.11
125	2.512	3.0	1.44	25	1.56
		1.5	0.36	75	1.43
					Avg 1.49 ± 0.07
130	2.481	3.0	1.44	12.5	1.26
		1.3	0.27	25	0.83
		1.0	0.16	50	0.90
		0.93	0.14	75	1.02
					Avg 1.00 ± 0.14

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Fig 1   Carbonyl absorbance area versus time during oxidation
of atactic polypropylene at 110°C

Fig 2   Carbonyl absorbance area versus time during oxidation
of atactic polypropylene at 120°C

Fig 3   Carbonyl absorbance area versus time during oxidation
of atactic polypropylene at 130°C

Fig 4   Carbonyl absorbance area versus time during oxidation
         of isotactic polypropylene at 120°C

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Fig 5   Carbonyl absorbance area versus time during oxidation
of isotactic polypropylene at 130°C.

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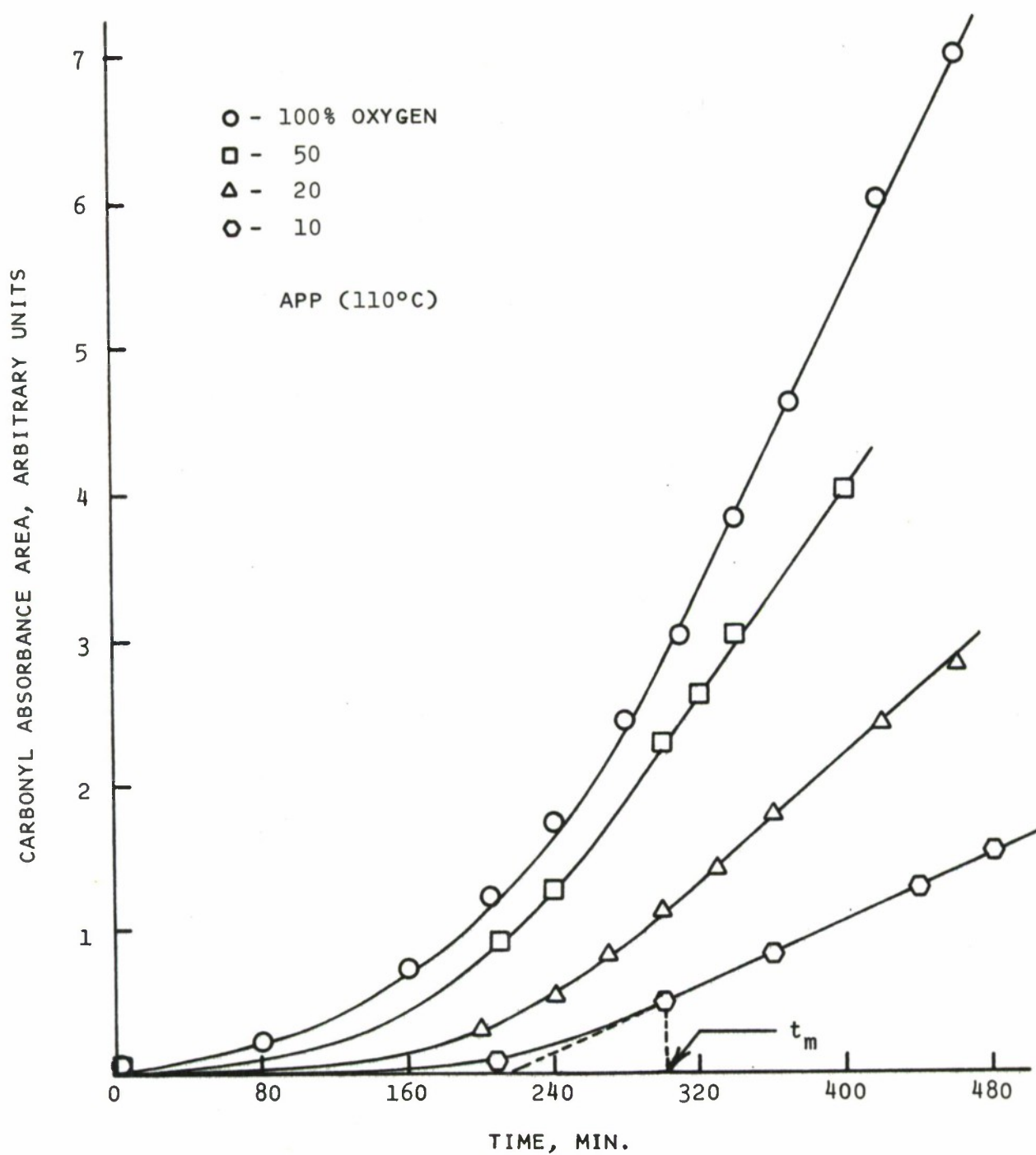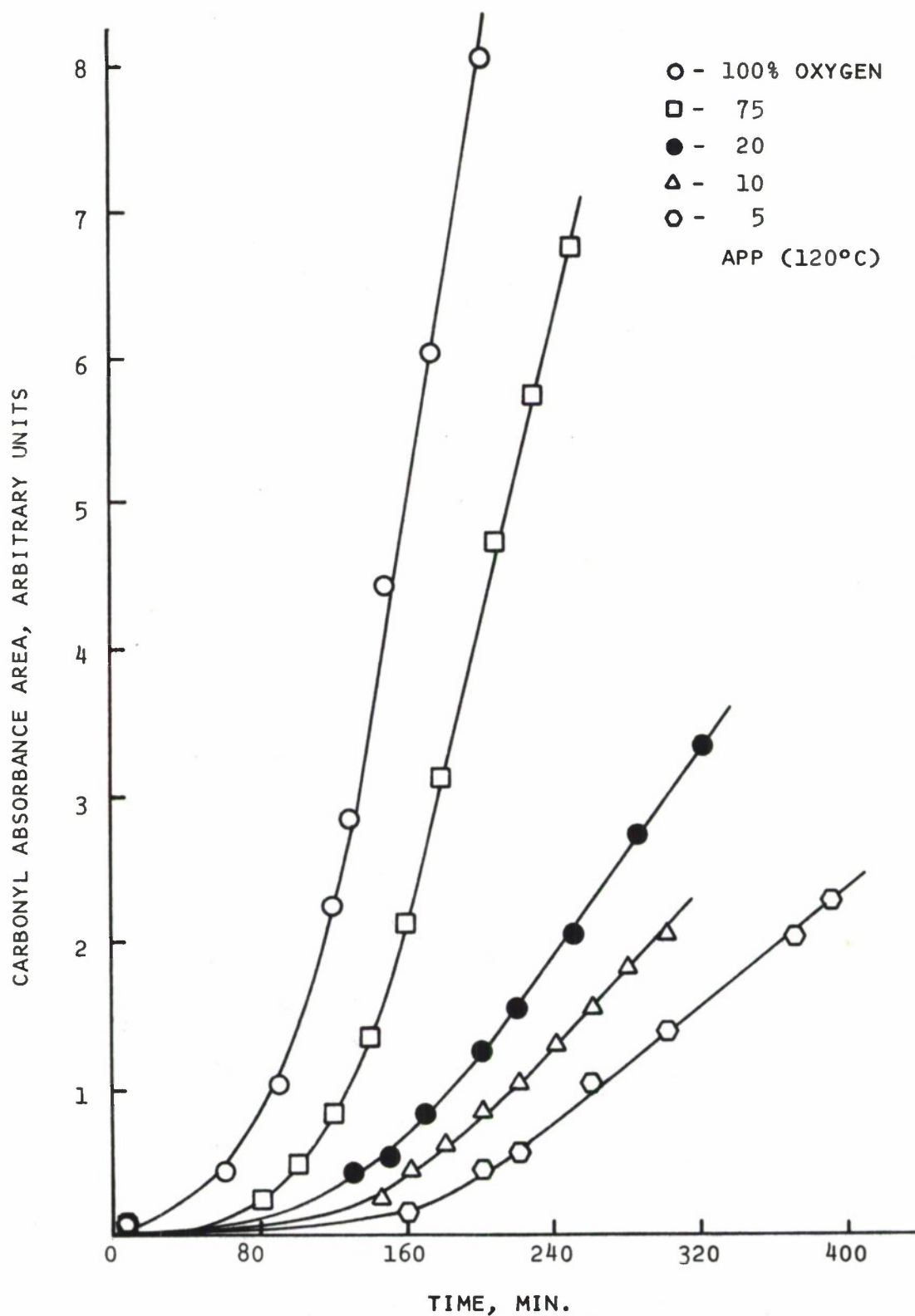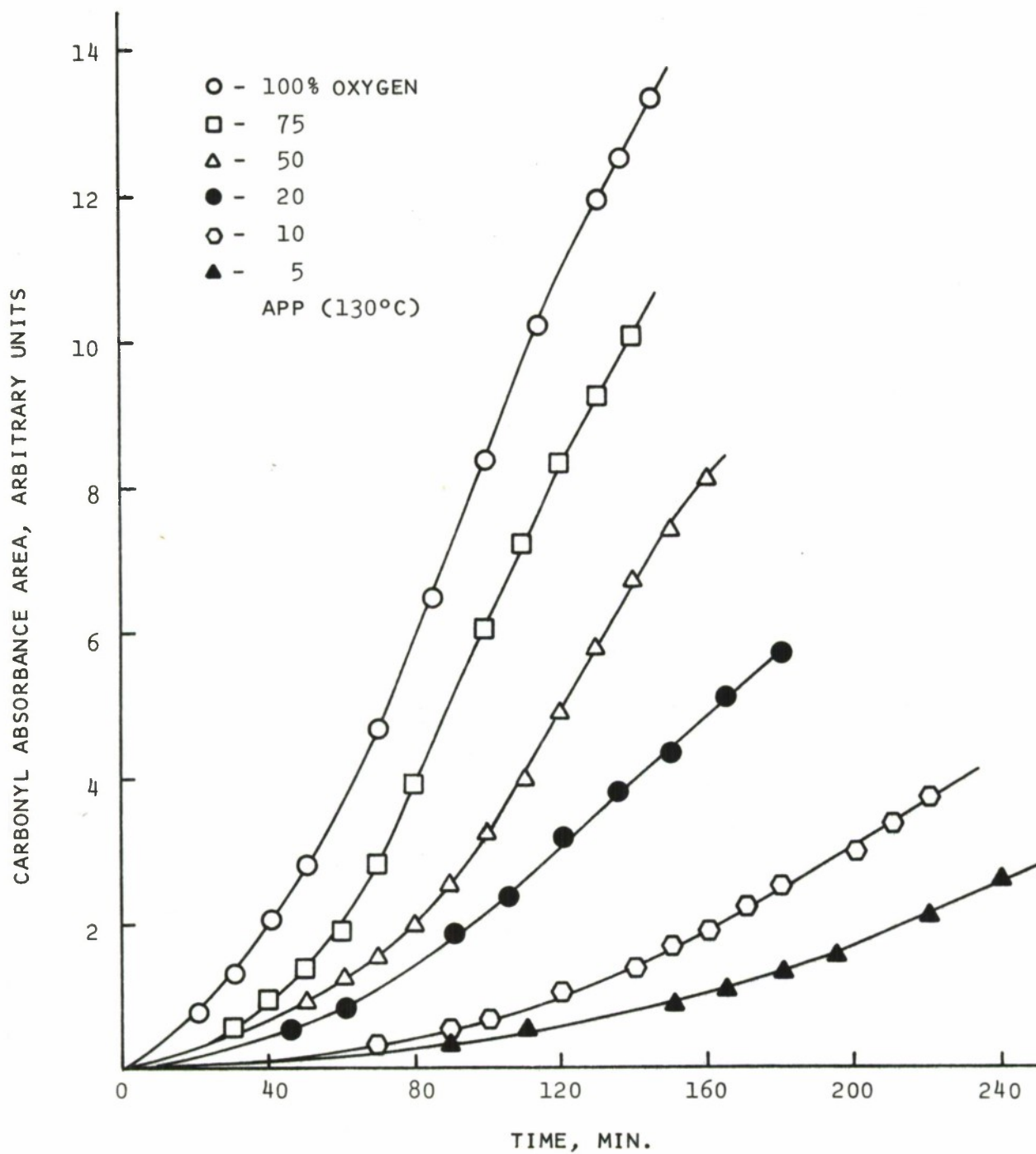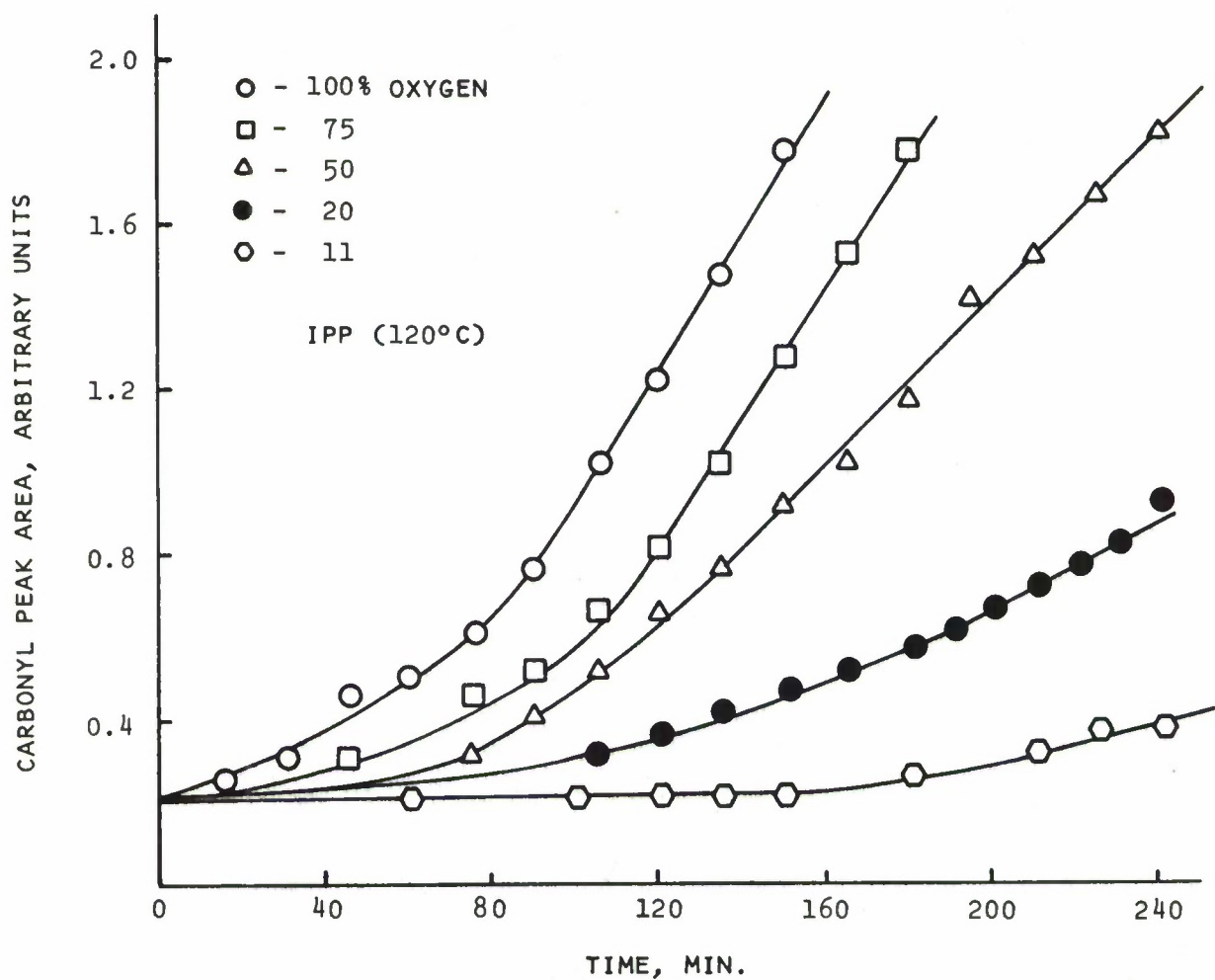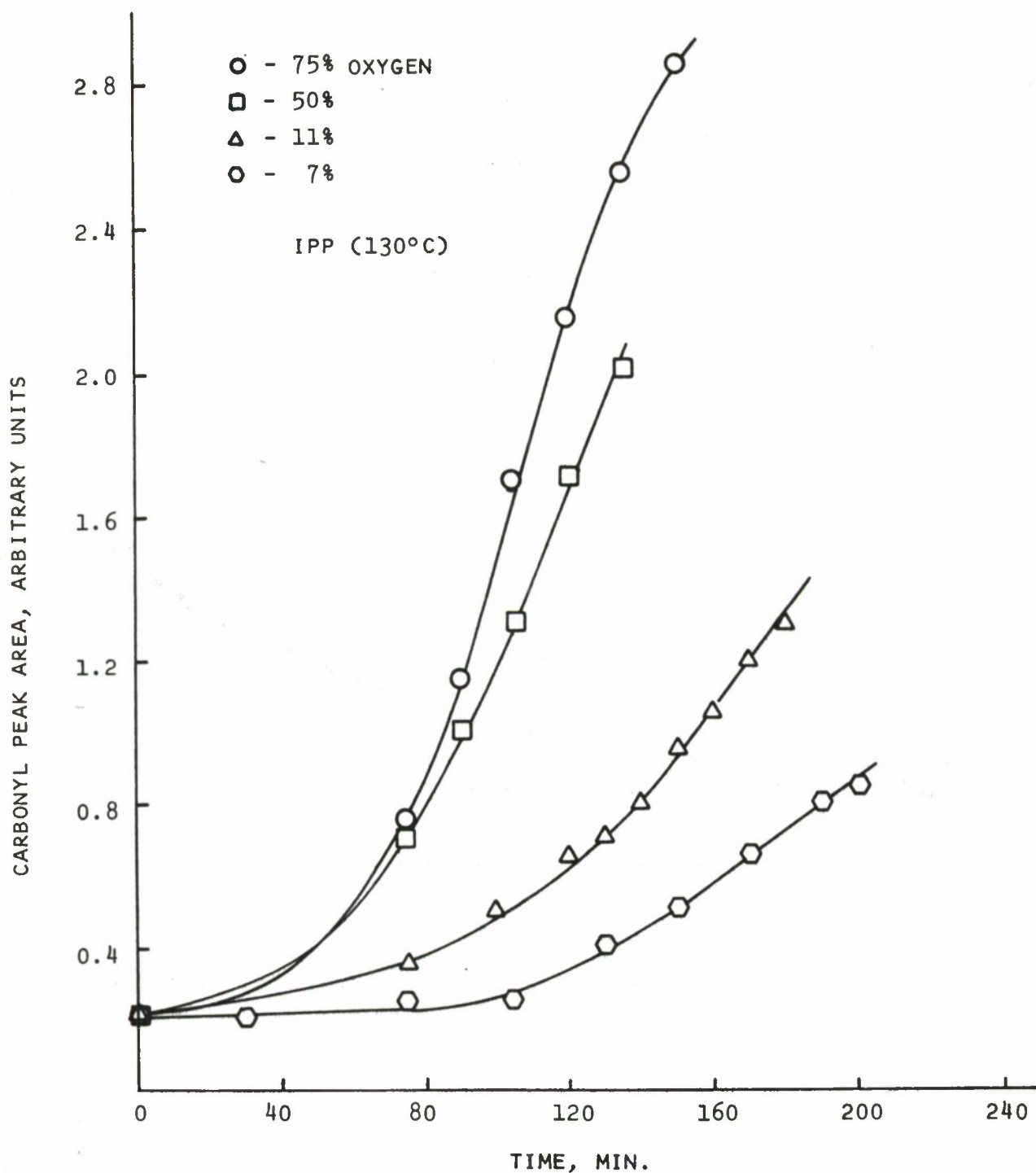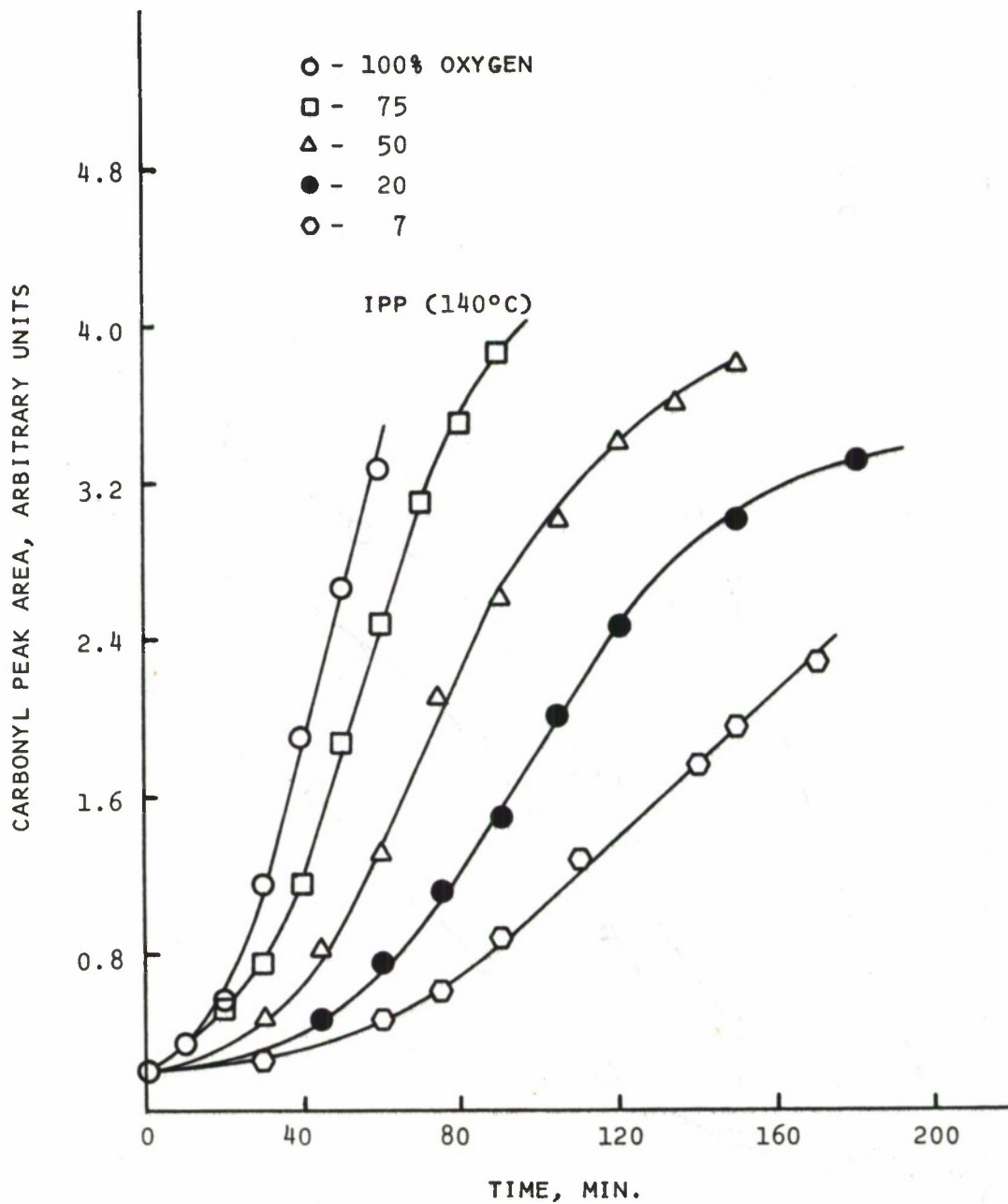Fig   6   Carbonyl absorbance area versus time during oxidation
of isotactic polypropylene at 140°C

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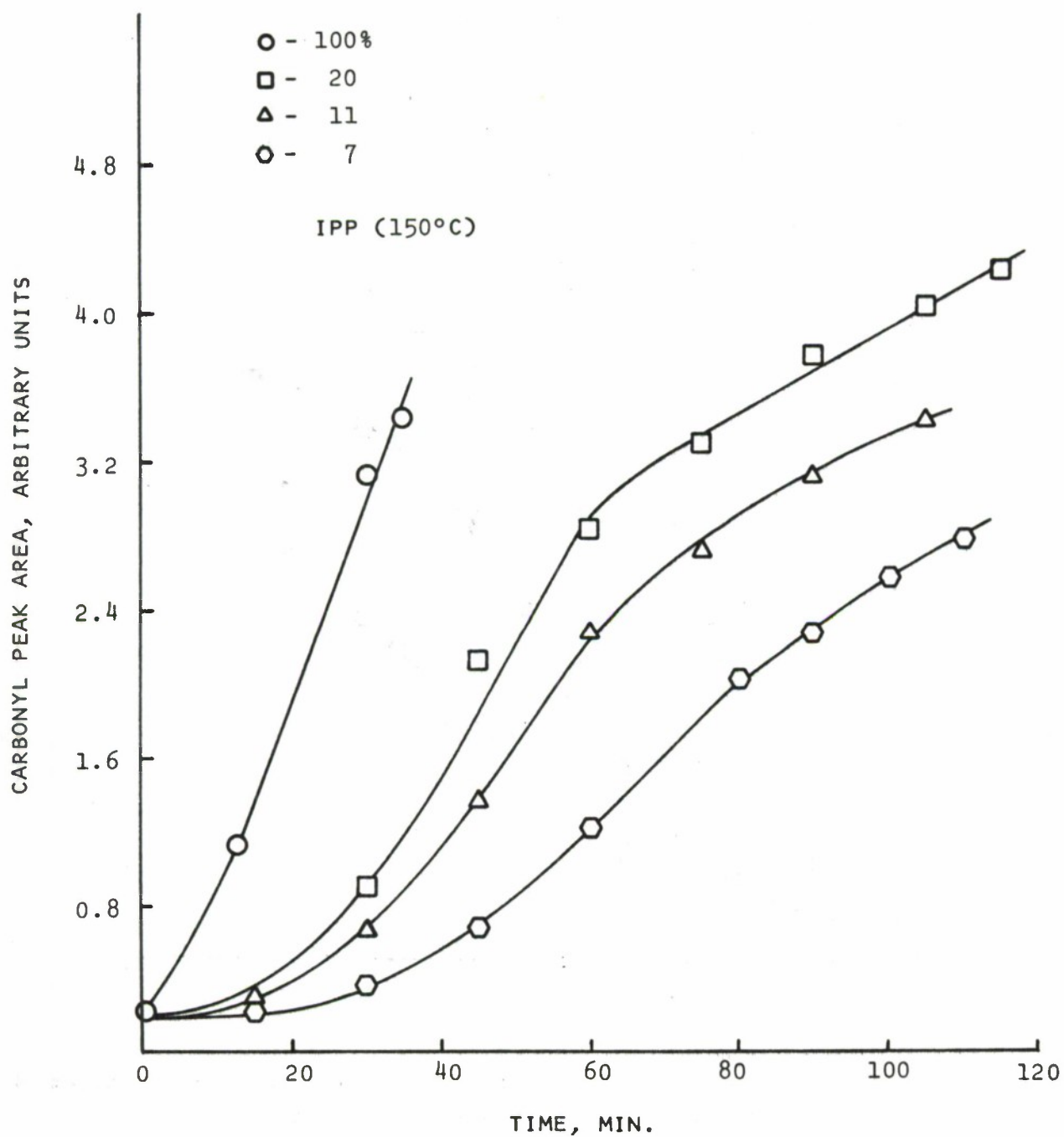
Fig 7 . Carbonyl absorbance area versus time during oxidation of isotactic polypropylene at 150°C

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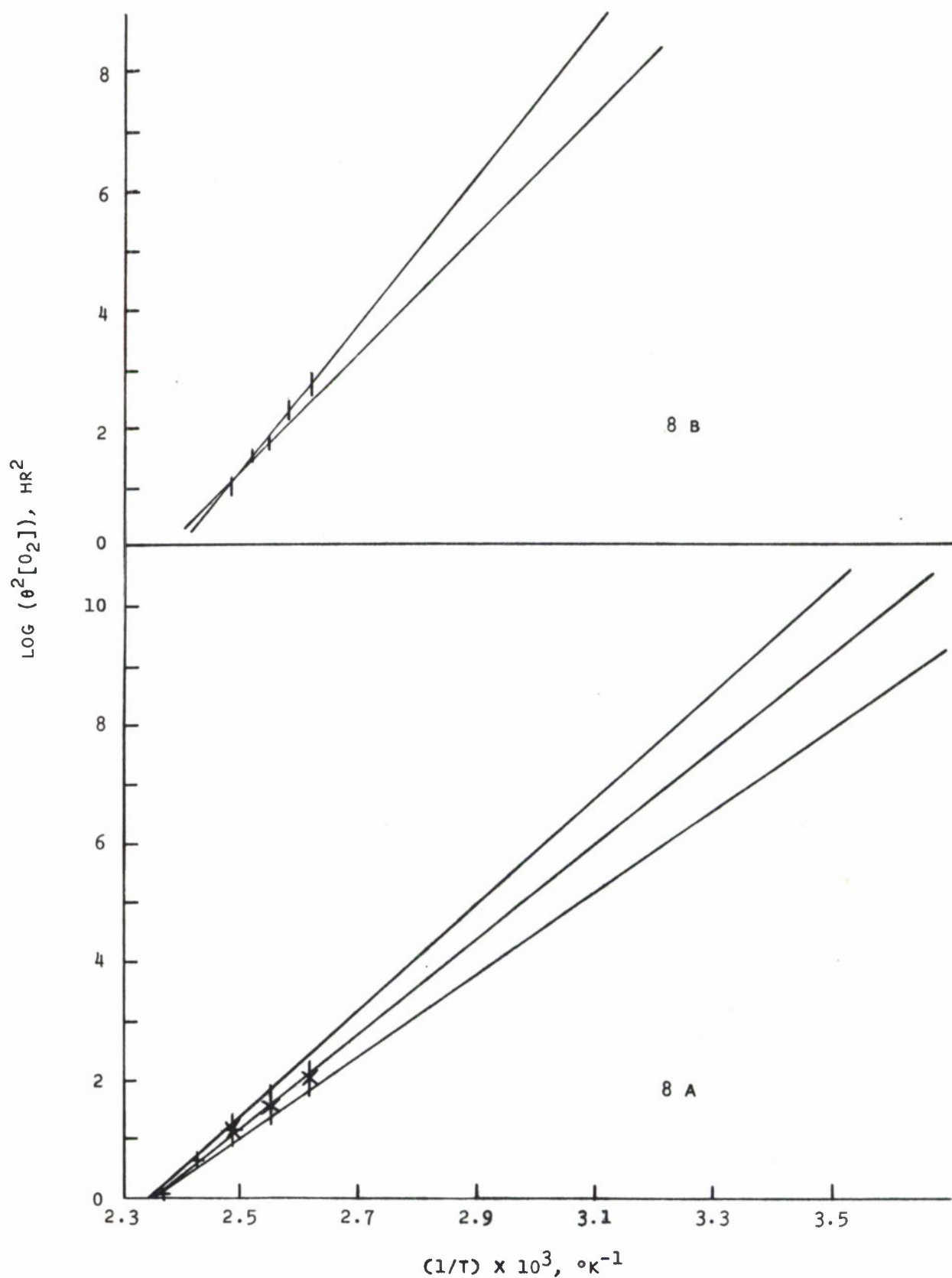
Fig 8. (A) log ($\theta^2 [O_2]$) versus reciprocal temperature for atactic and isotactic polypropylenes

(B.) log ($\theta^2 [O_2[$) versus reciprocal temperature for atactic polybutene-1

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Estimation of Shelf Life of Unstabilized Polyolefins from Thermal Oxidation Data

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13. ABSTRACT

Utilizing carbonyl formation data previously obtained during the thermal oxidation of unstabilized atactic polypropylene (APP), unstabilized isotactic polypropylene (IPP) and unstabilized atactic polybutene-1 (APB) along with a semi-empirical expression, it was possible to extrapolate and obtain arbitrary induction times (θ) at relatively low temperatures. Values of θ thus obtained were correlated with changes in physical properties, i.e., intrinsic viscosity and the product of ultimate elongation (UE) and ultimate tensile strength (UTS) (a product which is often referred to as the impact index).

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Shelf life						